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BAYER CROPSCIENCE SA *FR 2846325-A1

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Process for the preparation of 2,6-dihalo- 4-trifluoromethyl anilines, intermediates for phenyl pyrazole pesticides, by treatment of 4-trifluoromethyl aniline with a halogen

C2004-133004

Addnl. Data: BUATHIER B, LE ROY P

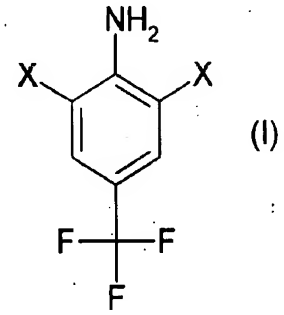
C(7-D8, 10-B4A) .2

NOVELTY

Process for the preparation of 2,6-dihalo- 4-trifluoromethyl anilines (I) by reaction of 4-trifluoromethyl aniline with a dihalogen.

DETAILED DESCRIPTION

Process for the preparation of 2,6-dihalo- 4-trifluoromethyl anilines of formula (I) by reaction of 4-trifluoromethyl aniline (II) with a dihalogen X_2, these being introduced simultaneously into a solvent in the molar ratio dihalogen/(II) of 1.9 - 2.5 and at a temperature of 100 - 300°C.



(I)

X = halogen.

USE

Intermediates for phenyl pyrazole pesticides, such as the insecticide fipronil.

ADVANTAGE

The process gives purer products than known methods, which are

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suitable for direct use in the manufacture of phenyl pyrazole pesticides.

EXAMPLE

Pure monochlorobenzene (12140 kg) was added to a 20 m^3 double envelope reactor under nitrogen, and heated to 110/oC. The reactor was then charged with 4-trifluoromethyl aniline as a 70 % solution in chlorobenzene and chlorine. The addition rates were 792 kg/h for the 4-trifluoromethyl aniline and 488 kg/h for the chlorine. Addition was continued for 6.5 hours, the temperature being kept at 110°C by cooling the vessel. On complete addition the residual content of 4-trifluoromethyl aniline and of chlorine was tested, and if any remained, sufficient of the other reactant was added to react with this residual reactant. Solvent was then removed from the vessel to leave (I, X = Cl) in 98.1 % yield. This product contained 0.05 % 4-trifluoromethyl aniline and 0.09 % monochloro-4-trifluoromethyl aniline as impurities.

TECHNOLOGY FOCUS

Organic Chemistry - Preferred Process (claimed): Preferably the reaction is effected using chlorine in a polar aprotic solvent, such as a chlorinated aliphatic solvent (dichloroethane), or a chlorinated aromatic solvent (chlorobenzene). The preferred molar ratio dihalogen/(II) is 2 - 2.05 and the reaction is preferably effected at 105 - 115°C.

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